



Europäisches Patentamt
European Patent Office
Office européen des brevets

(19)

(11) Publication number:

0 072 213
A1

(12)

EUROPEAN PATENT APPLICATION

(21) Application number: 82304144.7

(51) Int. Cl.³: C 09 K 17/00, C 05 G 3/04

(22) Date of filing: 05.08.82

(30) Priority: 07.08.81 GB 8124256

(71) Applicant: **UNILEVER NV, Burgemeester
s'Jacobplein 1 P.O. Box 760, NL-3000 DK Rotterdam (NL)**

(43) Date of publication of application: 16.02.83
Bulletin 83/7

(72) Inventor: **Bosley, John Anthony, 10 Philip Way Higham
Ferrers, Wellingborough Northamptonshire (GB)**
Inventor: **Dehnal, Roger Brian, 3 Laurence Court Dean
Street, Marlow Buckinghamshire (GB)**
Inventor: **Symien, Serge Alfred, 79 Queen's Drive,
Bedford (GB)**

(84) Designated Contracting States: **AT BE CH DE FR IT LI
LU NL SE**

(74) Representative: **Butler, David John et al, Unilever PLC,
Patent Division PO Box 31 Salisbury Square House
Salisbury Square, London EC4P 4AN (GB)**

(54) **Particulate compositions useful as plant growing media additives.**

(57) Superabsorbent gel compositions useful as plant growing media additives are prepared in particulate form by comminuting a partially-hydrated (10-80% moisture) gel mass in the presence of a finely divided flow aid, such as kaolin. The partially-hydrated granules are more easily mixed with damp compost or other growing media, and lead to improved soil properties, especially aeration, because the process gives rise to fewer fines which would otherwise tend to glue soil particles together. Preferred gels in foam form and based on polyacrylamide/acrylate are described.

ACTORUM AG

EP 0 072 213 A1

Z.411

PARTICULATE COMPOSITIONS USEFUL AS PLANT
GROWING MEDIA ADDITIVES

This invention relates to particulate compositions, especially water-retentive particulate polymeric compositions, useful as plant growing media additives.

5 Various forms of hydrogels ("superabsorbents") have been tested as, or at least proposed for use as, growing media additives, often referred to as soil conditioners, hydromulches, and soil-water retainers. Such additives can be used to improve the properties, such as water retention, of conventional plant growing media, for example soil, 10 peat, sand, loam, compost and sawdust. Alternatively, they can be used in conjunction with aqueous plant nutrient solutions to provide water retention and root support. An extensive review of the relevant literature is given by R A I Azzam, in Commun. Soil Science and Plant Analysis, 11(8), 767-834, (1980). It has however been difficult to 15 achieve the preparation of a product which has an adequately high degree of water uptake without adversely affecting soil properties such as aeration when the product is incorporated in soil to give it a higher water-holding capacity. 20

Moreover, we find that many of the available polymeric growing media additives do not hydrate readily without special precautions being taken, and moreover they contain clogging fines, require sieving, and can easily generate 25 further fines on handling.

77E21I

5 By the invention we provide a particulate composition useful as a plant growing media additive, comprising partially-hydrated water-retentive gel particles rendered free-flowing by the presence of a finely particulate flow agent.

10 The invention further provides a process for the preparation of such a composition, in which process a partially-hydrated mass of water-retentive gel is subdivided into particles in the presence of a finely particulate flow agent.

15 The gel particles are preferably rather coarse, ie at least 0.5 mm, preferably 1 mm or more typical particle size at 50-60% solids content (ie with the partially-hydrated gel containing 50-40% by weight water). The flow agent should be very much finer. Preferably the flow agent is hydrophilic, so that it does not hinder water uptake by the gel particles. Kaolin and fullers' earth are particularly preferred flow agents, but many alternative materials, such as fine talc powder, bentonite, fine aluminosilicates generally, and cellulose powders, can be used. Preferably 20 the flow agent is not affected by water contained in the gel, and preferably is non-hygroscopic. The gel particles are preferably free of significant amounts of water-soluble or swellable fines which could cause formation of a gluey product on further hydration or cause potential dust problems for users. For example, one desirable composition provided by the invention comprises no more than 3% by weight of gel particles finer than 0.5mm, and substantially no gel particles smaller than 0.25mm. Using the techniques 25 described herein, polymer fines in the 60-100 micron range can be avoided entirely, thus avoiding the need for sieving. This can preferably be achieved, in carrying out the invention, by avoiding milling the gel particles in the 30

dry state.

Preferably, much if not all of the flow agent is adherent to the surface of the gel particles. Indeed, in the ideal situation the flow agent forms an at least substantially continuous coating on all of the gel particles. Preferably, however, there should be little or no excess of flow agent present, as this could lead to dusting problems. The optimum level of flow agent present will clearly depend on the particle size of the gel, which determines the total surface area of the gel. In general, at least about 3%, and preferably at least about 5%, by weight of the flow agent will be required. The amount of the flow agent required is unlikely to exceed about 20% by weight, and an optimum level will typically be about 10%. These percentages are expressed by weight of the partially-hydrated gel product. Preferably the flow agent is added when the gel has a water content greater than about 16%, and more preferably greater than about 20% by weight, when it has a somewhat sticky surface, for example during comminution. For this purpose the water content of the gel should not exceed about 80% by weight, and need not in general exceed about 70% by weight, and an ideal level will generally lie in the range 30 to 60% by weight. The expression "partially-hydrated", as used in this specification in relation to the gel particles of the invention, implies water levels of roughly this order of magnitude, but it will be appreciated that different types of gels and flow agents may enable a partially-hydrated yet suitably free-flowing product to be made having a water content of greater than 70% by weight. To reduce the costs of handling and transport, preferably the partially-hydrated product should not contain more water than is necessary to achieve the benefits of the invention in this respect.

5 We find that such a product can be made with high
water-retentive capacity without substantially hindering
soil aeration or other agriculturally significant soil
properties eg gas-exchange facility, friability, drainage
and percolation properties, when the product is
incorporated as a conditioning water-holding agent.
Indeed, it is an advantage of the partially-hydrated
product of the invention that it can significantly improve
10 soil aeration under many circumstances, in contrast to many
other forms of "superabsorbent" gel products previously
tested for agricultural and horticultural purposes, which
have tended to lead to rather water-logged conditions.

15 To be of value in agriculture/horticulture as a
water-retentive additive, any gel product should be capable
of absorbing releasably at least 5, and preferably at least
10, times its own weight of water. Not merely can the gel
products of the invention achieve this, but we have been
able to produce free-flowing partially-hydrated particulate
20 gels capable of absorbing at least 15, generally at least
20, and often at least 25, times their dry weight of water
without significantly interfering with essential soil
properties. Indeed, some gel products of the invention are
capable of releasably absorbing 100 times, or more, their
dry weight of water. It will be appreciated that a typical
25 partially-hydrated product of the invention already
contains about 40% by weight of water. It will also be
appreciated that absorption figures for "pure" water may
not be particularly significant, because the presence of
even minute traces of ionised solutes can markedly affect
30 the water-absorbing properties of gel polymers. Hence it
is to be expected that any gel product will actually absorb
substantially less of an aqueous plant nutrient solution,
and so in practice their true absorbency is lower.
However, in general, the gel products of the invention are
35 capable of absorbing from 10 to 70 times their dry weight

of typical aqueous plant nutrient solution.

5 The manufacture of products of the invention can be relatively simple and convenient because a drying stage is not necessary: the gels can often be made with a water content equivalent to that of the final products.

10 Because their water content is at or near equilibrium, storage of the partially-hydrated gel products of the invention is facilitated, and they do not generally require sophisticated packaging to protect them from atmospheric moisture.

The products can be easily mixed with water or water-containing material, such as damp compost, without specially vigorous mixing, as is often required with fully dehydrated materials.

15 Even better aeration can often be achieved by products in which, as in a preferred embodiment, the partially hydrated gel composition has a foam structure preferably with a foam content equivalent to a volume increase of up to 100% or more, e.g. up to about 300%.

20 The invention is not limited to any restricted class or classes of water-absorbing and desorbing gel materials, and hence the invention can be beneficially applied to any of the superabsorbent gel types that have previously been used, or may be proposed for use, in an agricultural/
25 horticultural context. A wide variety of polymeric materials have been used to prepare superabsorbent gels, and these are reviewed, for example, in UK patent application No. 2,083,487A. Potentially, any hydrophylic water-swellaable polymeric material can be used, as long as
30 it is at least substantially water-insoluble. The polymer must have at least some ionic character to enable it to

absorb sufficient quantities of water. Some degree of cross-linking is generally essential to ensure low solubility. Examples of general classes of suitable polymers are polyacrylates, ie polymers and copolymers of acrylamide, acrylic acid, acrylic acid salts, acrylic esters and acrylonitrile; polysaccharide graft copolymers, ie starches and celluloses combined with polyacrylate chains, for example gelatinised starch-hydrolysed polyacrylonitrile graft copolymers; and cross-linked alkyl celluloses, such as carboxymethylcellulose cross-linked with epichlorohydrin. Any monomer can contain non-sterically-hindering substituents, eg methyl.

The gel product should have low deformability when fully hydrated, so that good soil aeration is maintained when it is added to or worked into soil and hydrated. Preferably the gel material is not significantly degradable, so that it retains its effect for a long time in a natural environment.

One very suitable example of such a polymeric material is a copolymer of acrylamide and acrylic acid salt cross-linked by inclusion of a minor proportion of a bifunctional cross-linking agent, preferably N,N'-methylene-bisacrylamide. In a particularly preferred embodiment of the invention, the gel is a cross-linked copolymer of acrylamide and acrylic acid salt, in a molar ratio of at least 70:30 but not greater than 95:5. Again, the preferred cross-linking agent is N,N'-methylene-bisacrylamide. Preferably the molar ratio of acrylamide to acrylic acid salt is at least 80:20 but preferably is not greater than 90:10. Methacrylates can be used.

The polymerisation and cross-linking conditions are preferably such as would (in the absence of cross-linking) yield substantially water-soluble high molecular weight

polymer, but includes enough cross-linking agent to reduce the amount of water-soluble polymer (which could lead to undesirable "glue" formation on hydration) to tolerable low levels, preferably less than 30% by weight of the dry polymer of the particulate compositions (ie less than about 20% of the partially-hydrated product). The level of cross-linking agent required will vary according to the types of monomers and cross-linking agents used, but generally the level of cross-linking agent will not need to be greater than 2%, and usually not greater than 1%, by weight of the total monomers. Usually the cross-linking agent will be present in an amount of at least 0.01% by weight of the total monomers. For the preferred acrylamide/acrylate copolymers used in the gel products of the invention, an ideal level of cross-linking agent will generally lie in the range 0.02 to 0.2% by weight of the total monomers. Examples of suitable cross-linking agents are ethylene glycol dimethacrylate, polyethylene glycol dimethacrylate, polyethylene glycol diacrylate, and most preferably 1,3,5-triacryloyl-hexahydro-S-triazine and N,N'-methylene-bisacrylamide. Mixtures of two or more cross-linking agents can be used if desired. The proportion of soluble material in the dry polymer can conveniently be regarded for present purposes as equal to the proportion by weight which becomes non-sedimenting and ultrafiltrable after agitation of particulate (uniformly ground through 1.5mm screen) polymer in 1% NaCl solution at room temperature for 2½ hours. This test can also be applied to the partially-hydrated product, with the omission of the grinding step.

It is preferred that the residual monomer in the polymer gel be not more than 1%, more preferably below 0.1%, most preferably below 0.05%. The special need for low monomer levels is due to the risk of toxicity to plants and users, and the levels which are acceptable can be

checked according to the nature of the monomer with this need in mind. This can be achieved for example either by ensuring complete enough polymerisation, and/or by heating the polymer to remove residual monomer, and/or by solvent-washing. For example, residual acrylamide monomer can be removed by heating in the range 90°C-150°C for a sufficient time.

Preferably a monomer solution is polymerised by conventional UV photoinitiation using any suitable initiator, such as a benzoin alkyl ether. The total monomer level will generally be at least about 20%, and preferably at least 25%, by weight of the solution. An upper limit will depend on the solubility of the monomers used and the viscosity of the solution, especially if a thin-film process is being performed. For acrylic monomers a solution concentration of up to about 55% by weight are very suitable, and an ideal concentration will generally lie in the range 30-50%. The initiation level can be varied over wide limits, eg 0.001 to 1% of the monomer, but for benzoin methyl ether, for example, about 0.1% of monomer weight is very suitable. Polymerisation conditions can be chosen to ensure autoacceleration by reaction heat to give short reaction times without causing excessive overheating.

Acrylic acid monomer units can conveniently be present as the potassium, sodium or ammonium salt or the salt of any other acceptable convenient counterion. When acrylic acid is used as a monomer it can be partly (e.g. 90%) neutralised by alkali (e.g. K_2CO_3) before the polymerisation reaction.

As mentioned above, according to a preferred feature of the invention, the polymer gel is in the form of a foam. Foaming can be produced for example by saturating the

reactant solution with a dissolved gas, and ensuring that the polymerisation conditions such that the exothermic polymerisation reaction causes a temperature rise sufficient to drive the gas reaction mixture to boil, after the reaction mix has become viscous, but before gelling is complete. In this way a bubble-containing polymer gel product is formed. As an example, a CO₂-effervescing system can be provided by saturating the reactant mix with a carbon dioxide, and/or using a carbonate to neutralise a monomer such as acrylic acid.

Foaming can be enhanced by the inclusion of a foaming agent (eg emulsifier, detergent) to the reactant mix prior to polymerisation, and this leads to particularly useful low-density cellular gel structures in the final product. A very wide selection of foaming agents can be used, the only likely restrictions being that the agent should not render the reactant solution opaque to UV light, should not have a harmful effect on plants if it were to be leached from the gel during use, and relative cost (although the level of foaming agent employed would in any event be very low). Preferably the quantity of foaming agent added to the reactant mix should be just sufficient to give the desired degree of foaming during the polymerisation, but this quantity will clearly vary from one agent to another. Very generally, it can be said that the quantity needed will lie in the range 0.01 to 0.1 gm/litre of reactant solution. Examples of suitable foaming agent that we have found to be effective are soaps, such as sodium stearate, and emulsifiers, such as sodium stearyl-2-lactylate. Examples of other commercially-available foaming agents are hydrolysed proteins, such as "Hyfoama DS" (Food Industries NV) and glycerol esters, such as "Emulsponge 2002" (PPF International).

Foamed products made in this way are especially

5 advantageous in that they possess a "two-stage"
water-holding capacity, by virtue of water taken into cells
formed by the effervescence and water physically absorbed
in the microporous polymer gel structure itself. They also
can show increased water uptake rate, because of their
greater surface-to-volume ratio.

10 Particulate gel compositions according to embodiments
of the invention can conveniently be made by dividing a
partially-hydrated water-absorptive polymer gel mass, e.g.
made as described above, using a knife mill, or other
suitable comminution device which has a shearing/cutting
action at a relatively slow speed, eg about 500 r.p.m. A
device having a rotary-bladed scissors-type
shearing/cutting action is preferred, and a good example is
15 the "Blackfriars" (TM) cutter.

20 The presence of a flow aid is important here to
prevent cut gel pieces from adhering to each other and to
the comminution device. As fines are to be avoided if
possible, average particle sizes preferably are of the
order of 0.5 to 5 mm, and preferably substantially no
particles of gel are below 125 microns. The size of the
particles is given as for the partially-hydrated condition,
as the particles generally increase in size on hydration.
We find that this method of comminution of a
25 partially-hydrated material using a knife or cutting mill
helps to avoid any need to sieve the gel product.

30 According to the invention, the particulate gel
compositions so produced can be added to soil to improve
its water-holding ability, or to give water-retentive
layers, mulches and packaging for use in horticulture and
agriculture. It will be appreciated that the gel
composition itself will not provide any nutrients to
sustain plants, and it must therefore be regarded primarily

as a moisture-retentive and physically-supportive material. When used in combination with an aqueous nutrient solution (containing e.g. phosphates, nitrates, plant hormones and growth regulants) and/or one or more traditional plant growing media, such as peat, loam, compost or moss, the whole provides a very suitable material in which plants can germinate, root or grow. The gel compositions of the invention are particularly well suited for use in the potting or rooting of immature plants in a nursery prior to transfer to field or plantation conditions, and as such can be mixed in minor proportion with a rooting (root-holding) medium such as wood fibre, peat, forest bark, straw, loam, or particulate plastics material.

The proportion of gel product used can be varied according to the situation. When employed as a soil additive, levels of inclusion as low as 0.5-1% by weight of gel/volume of growing medium (assessed in terms of the partially-hydrated gel product) can lead to significant soil improvements. In this context generally only a few percent of the gel are required, and levels in excess of 4% by weight may not be considered economic. However, in a small-scale potting or rooting procedure the gel can be used simply in conjunction with an aqueous nutrient solution and the gel therefore represents 100% of the solids present, although in this context it will generally be sensible on cost grounds to mix the gel with one or more cheaper supportive fillers such as sawdust, straw or sand.

The invention is illustrated without limiting its scope, by the several features of the following Examples.

Example 1

Preparation of a flowable particulate composition suitable for agricultural or horticultural use.

5 Acrylic acid (950 g) containing methylenebisacrylamide (10 g) was neutralised either with potassium hydroxide (700 g) or potassium carbonate (863 g) as a solution in water (1250 ml). The temperature of the mixture was kept below 30°C during the neutralisation. Benzoin methyl ether (1.0 g) was dissolved in acrylic acid (50 g) and this solution was added to the mixture. The solution was shielded as much as possible from light. The whole was purged with nitrogen.

10 The reactant mix was then pumped to give a film (film thickness varied from 3-10 mm) on a moving conveyor shaped by the action of folding cheeks and a suction box to give a trough shape configuration which contained the liquid reactants. The conveyor then carried the solution through
15 a nitrogen filled cavity where it was irradiated with long wave-length UV light (2 x 40 watt tubes peaking at 365 nm, giving 3200 $\mu\text{W}/\text{cm}^2$ incident light intensity). The solution rapidly gelled to give a rubbery polymer. The irradiation time was typically in the range 6-9 minutes.

20 The partially hydrated gel was minced in a continuous process in a "Blackfriars" (TM) cutter with the addition of at least 5% by weight fine-particle-size (40-60 μm) kaolin, fuller's earth or bentonite, to give a chip product in which 97% of the particles had a particle size greater than
25 0.6mm and less than 2mm, and substantially no fines. The product had a moisture content about 40% by total weight, a bulk density of about 0.6g/ml, and a proportion of soluble polymer (by reference to the total dry polymer) of 22%, when measured as described above. The product could be
30 dried but should not be milled when dry, to avoid making fines. The product absorbed about 20 times its own weight of a conventional aqueous phosphate-containing plant nutrient solution.

The product was suitable for use in a mulch, as a soil additive or in a mixture with non-soil components, eg straw or peat, for covering or enclosing plant root systems.

Example 2 (Foamed gel crumbs)

5 Acrylic acid (950g) containing methylenebisacrylamide
(10g) was (c. 90 mole %) neutralised by addition of
potassium carbonate (862g) as a solution in water (1200g).
The temperature of the mixture during the neutralisation
process was maintained at 20°C or less. Benzoin methyl
10 ether (1g) in acrylic acid (50g) was added to the
neutralised acrylic acid solution followed by a thermal
initiator 2,2'-azobis(2-amidinopropane) hydrochloride (1g)
as a solution in water (50g). The reactant mix was
shielded as much as possible from light.

15 The solution was poured into a glass dish to give a
film thickness of between 5 and 7 mm and was irradiated by
long wave length ultra-violet light (peak wavelength 365nm,
incident light intensity 3200 $\mu\text{W}/\text{cm}^2$). Polymerisation
was rapid and accompanied by a rapid temperature rise, the
20 temperature reaching a maximum of approximately 115°C after
2-3 minutes. There was a sudden and large increase in the
volume of the polymer, due both to the boiling point of the
aqueous mixture being exceeded and to the release of carbon
dioxide from solution. This resulted in a highly foamed
25 rubbery polymer. The total irradiation time was 8 minutes.

 After cooling the foamed polymer was removed from the
glass tray and comminuted in a "Blackfriars" (TM) cutter
with the addition of at least 5% by weight fine particle
size (40-60 μm) fuller's earth, kaolin or bentonite to give
30 a crumb product where 97% of particles lay in the size
range 0.6-2.0 mm and with substantially no fines. The
moisture content of the product was 36%, the bulk density

5 about 0.5g/ml and the proportion of soluble polymer when measured as described above up to about 30% (of the dry solids, or about 20% by weight of the partially-hydrated product). The partially-hydrated product was able to absorb about 17 times its own weight of a conventional aqueous phosphate-containing plant nutrient solution.

Example 3 (Polyacrylamide copolymer)

10 Acrylic acid (48.7g) containing benzoin methyl ether (0.24g) was completely neutralised with potassium carbonate (47.3g) as a solution in water (120 mls). To this mixture acrylamide (192g) and N,N'-methylene-bisacrylamide (0.24g), as a solution in water (240 mls), was added. The solution was shielded from light as much as possible and purged with nitrogen for 10 minutes.

15 The reactant mix was poured into pyrex glass dishes so as to give a film thickness of approximately 7.5mm and irradiated with long wave length ultra-violet light (2 x 40 watt tubes peaking at 365 nm giving an incident light intensity of $4000 \mu\text{W}/\text{cm}^2$). The solution rapidly gelled to a give a rubbery polymer accompanied by a rapid temperature rise, typically from 20°C to 100°C in 1½ minutes. The total irradiation time was typically 5-10 minutes. The resulting material was not foamed.

25 The rubbery polymer film was removed from the glass dishes and is comminuted in a "Blackfriars" (TM) cutter with the addition of at least 5% by weight fine particle size (40-60 μm) cutting aid such as fullers' earth, kaolin or bentonite to give a chip product where 97% of particles lay in the range 0.6-2.0 mm and with substantially no fines. The moisture content of the product was about 43%, the bulk density about 0.7g/ml and the proportion of soluble polymer when measured as described above was about

30

13% (of the dry solids, or about 9% by weight of the partially hydrated product). The partially-hydrated product could absorb about 27 times its own weight of a conventional aqueous phosphate-containing plant nutrient solution.

Example 4

To a solution of acrylamide (1800g) in water (1950 mls) was added potassium carbonate (195.5g) in water (500 mls). To this mixture acrylic acid (204 g) containing benzoin methyl ether (2 g) and N,N'-methylene-bisacrylamide (1 g) is slowly added with stirring to complete the neutralisation reaction. The solution was shielded from light as much as possible and purged with nitrogen to flush out carbon dioxide from the carbonate neutralisation.

The reactant mix was then pumped to give a film thickness of 5-9 mm on a moving conveyor shaped by the action of folding cheeks and a suction box to give a trough shape configuration which contained the liquid reactants. The conveyor carried the solution through a nitrogen-filled cavity where it was irradiated with long wavelength UV light (2 x 40 watt tubes peaking at 365 nm, giving $3500 \pm 200 \mu\text{W}/\text{cm}^2$ incident light intensity). Polymerisation was rapid and accompanied by a rapid temperature rise, reaching in excess of 100°C after 2-3 minutes. Some large bubbles were formed in the polymer film due to the boiling point of water being exceeded, but the material is not foamed. The total irradiation time was in excess of 20 minutes.

The rubbery polymer film was cooled and comminuted on a "Blackfriars" (TM) cutter with the addition of 5% by weight of fine particle size cutting aid (fullers' earth). The moisture content of the product was 41%, the bulk

density 0.6 g/ml and the soluble fraction of the partially-hydrated product was 6% (equivalent to about 9% by weight of the dry polymer). The product absorbed about 20 times its own weight of a plant nutrient solution.

Example 5

5

The procedure of Example 4 was repeated, using identical materials, quantities and reaction conditions, except that a dispersion of "Emulsponge 2002" (a commercially-available foaming agent obtained from PPF International) was added to the reactant mix and the nitrogen purging was omitted. The dispersion contained 1 g of foaming agent in 50 ml water.

10

During the polymerisation reaction there was a sudden and large increase in the volume of the polymer, due both to the boiling of the aqueous mixture and to the release of carbon dioxide from solution. The formation of a foamed gel was greatly enhanced by the addition of the foaming agent.

15

The foamed polymer film was cooled and comminuted on a "Blackfriars" (TM) cutter with the addition of 5% by weight of fine particle cutting aid (kaolin). The moisture content of the product was 41%, the bulk density 0.5 g/ml and the soluble fraction of the partially-hydrated product was 7% (equivalent to about 11% by weight of the dry polymer). The product absorbed about 25 times its own weight of a plant nutrient solution.

20

25

All the products described in Examples 1 to 5, had a non-powdery look and feel, and a friable, granular, although soft, texture to the touch, and were well compatible with soil and other plant growing media.

30

Example 6

The partially-hydrated gel product of Example 4 was used in plant growing trials as follows:

5 The gel product was used in conjunction with three separate growing media, sphagnum peat, coarse sand and good loam, each supplemented by a commercially-available controlled-release fertiliser. The trials were conducted in plastics plant pots each having a top diameter of 14 cm and a depth of 9 cm, packed with 600 cc of growing media. The gel product was included at levels of 0.5, 1.0 and 2.0% wt/vol, and each growing media without any gel was used as a control. There were four pots of each combination, giving a total of 48 pots. Each pot contained 0.84 gm of the controlled-release fertiliser "Osmocote". The prepared pots were filled with water and drained for 24 hours prior to seed sowing.

10 To ensure uniformity of plant material, Phaseolus vulgaris seeds of the cultivar PROVIDER were chitted and sown five to a pot. The plants were grown for the five weeks under glass until the formation of the first flower buds, at which time plant height, number of flowering nodes and dry weight of the aerial portion were recorded. Watering throughout the growing period was by means of capillary matting. The results are given in Table 1, and indicate that the addition of the gel induced increases in plant height and number of nodes, and statistically significant increases in dry weight, in all media.

Table 1

Influence of gel on the growth of P.vulgaris in three growing media.

Medium	Gel (% wt/vol)	Plant height (cm)	Flowering nodes	Dry weight (gm)
Sand	-	25	1	3.812
	0.5	26	2	5.152
	1.0	26	2	4.112
	2.0	30	2	4.890
Peat	-	36	2	8.497
	0.5	42	3	9.170
	1.0	47	3	10.060
	2.0	47	3	10.062
Loam	-	37	1-2	4.785
	0.5	40	2	6.125
	1.0	43	3	7.185
	2.0	40	2	6.897
Sig. difference (P: 0.05)				0.968

Example 7

The use of gel additive in sapling production

The partially hydrated gel product of Example 4 was used in growing trials to raise two species of tree from seed.

5 The gel product was used in conjunction with a peat based potting compost supplemented with additional fertiliser. The gel product was included at 0.5, 1.0 and 2.0% wt/vol and growing medium without gel was used as control.

The compost contained the following additional fertiliser (per m³):

10 2.0 kg "Osmocote" slow-release fertiliser
(8-9 month formula)
0.2 kg trace elements
0.2 kg ferrous sulphate heptahydrate
3.0 kg magnesian limestone
0.5 kg single superphosphate

15 "Japanese paper pots" (for sizes, see below) were filled with growing medium allowing some space for the swelling of the gel product on addition of water. For each medium mixture under test, 20 pots per tree species were prepared.

20 Alders (Alnus glutinosa) were sown at 1 seed per pot (pot size: 4.5 cm dia, 7.5 cm deep) and covered with 3-5 mm of lime-free sand. Oaks (Quercus petraea) were sown at one chitted seed per pot (pot size: 4.5 cm dia, 15 cm deep) at a depth of approx. initially 2.5 cm. The pots were then thoroughly watered and covered with polyethylene sheet to prevent drying out. The sheet was removed at
25 approximately 10% emergence.

Germination and emergence counts were made at 14 and 28 days. These are shown in Table 2.

Table 2

<u>Numbers of emergent seedlings</u>									
	14 days				28 days				
	0	0.5	1.0	2.0%	0	0.5	1.0	2.0%	
<u>Gel</u>									
<u>Alnus</u>	9	15	17	18	14	15	20	19	
<u>Quercus</u>	3	8	9	13	17	13	20	20	

The plant heights were recorded after 9 weeks growth and are given in Table 3.

Table 3Average plant heights at 9 weeks (in mm)

<u>Gel</u>	0	0.5	1.0	2.0%
<u>Alnus</u>	**	24.0	36.2	45.6
<u>Quercus</u>	50.2	59.7	76.6	94.8

5

** although the original seedling emergence was high all the controls were subsequently lost through desiccation. The presence of gel product greatly reduced these losses, the numbers of surviving plants were 1, 9 and 12 respectively for the three treatments.

5 The plants were then potted on into 2.5 litre polyethylene containers using the same compost mixture and gel treatments except that the gel was swollen by the addition of approximately 20 times its original weight of aqueous nutrient solution prior to mixing with the compost. Plant heights were recorded at 19 weeks after the original sowing date.

Table 4

<u>Average Plant heights at 19 weeks (in cm)</u>				
Gel:	0	0.5	1.0	2.0%
<u>Alnus</u>	-	53	44.1	59.3
<u>Quercus</u>	19	27.4	29.6	36.5

10 Example 8

Effect of incorporation of gel
additives on growing media phase volumes

15 The gel product of Example 4 was added to a growing medium as detailed in Table 5. The mixture was placed in a 10 cm diameter plant pot and placed on capillary matting saturated with a standard concentration of an aqueous plant nutrient solution. When the contents of the pot had reached equilibrium the phase volumes of the constituents (air, water, solid) were calculated using absolute
20 densities obtained using a helium-air pycnometer. The values are expressed as air:solid and water:solid ratios.

Percentages of gel product added are weight/volume, eg 1% represents 1g additive in 100 mls medium.

Table 5

	<u>Medium</u>	<u>A/S</u>	<u>W/S</u>
	Peat based potting compost	2.1	7.1
	+ ½% gel product	2.5	7.4
5	+ 1% "	2.9	7.9
	+ 2% "	3.6	8.7
	Compost tree bark	3.6	5.5
	+ ½% gel product	4.2	6.6
	+ 1% "	4.5	6.8
10	+ 2% "	4.5	7.2

As can be seen from the Table 5, the addition of water absorbent polymer improves both the air and water holding properties of the growing media as reflected in their air:solid and water:solid ratios.

Example 9

Example 8 was repeated except that:

- i) the pots were covered tightly with polyethylene sheet to prevent moisture loss from the surface of the growing medium.
- 20 ii) the gel additive was swollen by addition of approximately 20 times its own weight of an aqueous plant nutrient solution prior to mixing with the growing medium.

The results are show in Table 6:

Table 6

<u>Medium</u>	<u>A/S</u>	<u>W/S</u>
Peat based potting compost	5.0	8.6
+ 1% gel product	5.6	9.9
Composted tree bark	5.1	5.7
+ 1% gel product	5.4	6.2

The increase in air:solid and water:solid ratios show that the air and water holding properties of the growing media have been improved.

* * * * *

CLAIMS

1. A particulate composition useful as a plant growing media additive, comprising partially-hydrated water-retentive gel particles, having a water content greater than about 16% but not greater than about 80% by weight, rendered free-flowing by the presence of a finely particulate flow agent.
2. A composition according to claim 1. wherein the flow agent is kaolin or fullers earth.
3. A composition according to either of the preceding claims, wherein the flow agent comprises at least about 3% but not more than about 20% by weight of the composition.
4. A composition according to any one of the preceding claims, wherein the polymeric material is a cross-linked copolymer of acrylamide and (meth)acrylic acid salt.
5. A composition according to Claim 4, wherein the molar ratio of acrylamide to (meth)acrylic acid salt in the copolymer is at least 70:30 but not greater than 95:5, and the copolymer is cross-linked such that the proportion of water-soluble polymer is less than 30% by weight of the dry polymer material.
6. A plant growing composition, comprising a particulate composition according to any one of the preceding claims together with a conventional growing medium such as soil, peat, loam or compost.

7. A process for the preparation of a particulate composition useful as a plant growing media additive, in which process a partially-hydrated mass of water-retentive gel, having a water content greater than about 16% but not greater than about 80% by weight, is subdivided into particles in the presence of a finely particulate flow agent.
8. A process according to Claim 7, wherein the gel mass has a water content greater than about 20% by weight.
9. A process according to Claim 7 or Claim 8 wherein the gel mass is rendered particulate by means of a comminution device having a shearing/cutting action.
10. A process according to Claim 9, wherein the comminution device is a knife mill.
11. A process according to any one of Claims 7 to 10, wherein the flow agent is kaolin or fullers earth.
12. A process according to any one of Claims 7 to 11, in which the gel mass is produced by copolymerising an aqueous reactant mix containing acrylamide and (meth) acrylic acid salt.
13. A process according to any one of claims 7 to 12, in which a mixture of acrylamide and (meth)acrylic acid salt in a molar ratio of at least 70:30 but not greater than 95:5 is copolymerised in the presence of a cross-linking agent in an amount sufficient to ensure that less than 30% of the resulting polymeric composition (expressed by weight of the composition when dry) is water soluble.

14. A process according to Claim 13 wherein the cross-linking agent is 1,3,5-triacrylol-hexahydro-S-triazine and/or N,N -methylene-bisacrylamide.
15. A process according to Claim 13 or Claim 14 wherein the quantity of cross-linking agent present in the reactant mix is from 0.02 to 0.2% by weight of the total monomers.
16. A process according to any one of Claims 13 to 15, wherein the resulting polymer is produced in foam form by causing a gas to be driven from the reaction mix after it has become viscous but before gelling is complete.
17. A process according to Claim 16, in which the gas is carbon dioxide.
18. A process according to Claim 16 or Claim 17, in which foaming is enhanced by the presence of a foaming agent.

0072213



European Patent
Office

EUROPEAN SEARCH REPORT

Application number

EP 82 30 4144

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. ³)
A	<p>--- US-A-3 437 625 (B.BONNEL) *Column 1, lines 41-65; column 2, lines 1-10; claims 1-4*</p>	1	<p>C 09 K 17/00 C 05 G 3/04</p>
A	<p>--- US-A-3 557 562 (H.C.McLAUGHLIN) *Column 2, lines 13-65; claims 1-3, 12, 23, 24*</p>	1, 2, 14	
A	<p>--- EP-A-0 023 347 (ISAFLEX) *Page 1, lines 1-12; page 3, lines 21-27; claims 1-4*</p>	1, 6, 14	
A	<p>--- US-A-2 856 380 (R.W.ROTH) *Column 1, lines 15-26, 44-66; column 2, lines 13-72; column 3, lines 13-75; column 4, lines 1-61; claims 1-10*</p>	1, 14	
A	<p>--- US-A-3 857 991 (HIGASHIMURA) *Column 2, lines 25-68; column 3, lines 1-52; claims 1, 3, 4*</p> <p>-----</p>	1, 4, 14	<p>TECHNICAL FIELDS SEARCHED (Int. Cl. ³)</p> <p>C 09 K C 05 G</p>
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 05-11-1982	Examiner BOULON A.F.J.
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</p>			

EPO Form 1503 03 82